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Role of Methyl Ester (Biodiesel) Production by Synthesized Nanocatalyst: Analysis of Product, Properties, Composition, and Cost Estimation

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Abstract

One alternative energy strategy involves recycling used cooking oil (UCO) to create biodiesel, a liquid fuel that can be used in various applications. There are several advantages, including financial, environmental, and waste management, to making biodiesel from UCO and methanol using a calcium oxide (CaO) nanocatalyst. In this research, CaO nanoparticles are synthesized using the sol-gel process and characterized using X-ray diffraction (XRD) and scanning electron microscope (SEM) techniques to improve this generation of biodiesel from UCO. At the laboratory scale, the methyl ester production reaction parameters were optimized. At 50 °C, a 1:8 UCO oil-to-methanol ratio, a 1% by-weight catalyst loading rate, and a 90-min reaction duration, the highest biodiesel yield. The characteristics of biodiesel characterized using Fourier transform infrared (FTIR) spectroscopy and gas chromatography-mass spectrometry (GC-MS) analysis were also evaluated per the American Society for Testing and Materials (ASTM D6571). The biodiesel cost estimation was calculated in Indian Rupees (INR) and United States Dollars (USD). The final cost estimation was 45.23 INR, equivalent to 0.55 USD per liter; it was an economical process with high product value.

Keywords

Biodiesel, Nanocatalyst, Transesterification, Used cooking oil, Methyl ester, Cost analysis

Introduction

An essential resource that serves as a gauge for a country's degree of development is energy. Most of the home, commercial, and transportation operations rely on the availability of energy. With the availability of hydrocarbons, energy is taken from petrochemical sources like coal, oil, and natural gas, which are problematic for the environment. Burning fossil fuels poses many health dangers to the public and environmental issues that could have long-lasting effects on global warming [1, 2]. Renewable energy includes wind, hydro, solar, biomass, and biofuels. Developing sustainable alternative energy sources that are biodegradable, environmentally benign, non-toxic, and release fewer aromatic hydrocarbons, SO_x, NO_x, CO, and other greenhouse gases is essential. Biodiesel has gained a lot of interest as an environment and eco-friendly substitute for petroleum products [2, 3]. Using several catalysts, Biodiesel can be created from different oils, including castor, sunflower, soybean, and palm. There is mounting evidence that biodiesel offers several desirable properties. It has low toxicity, can be recycled, is naturally lubricous, contains almost no sulfur, and can be used in most engines with minor modifications [3]. It can be combined with petrol diesel in various ratios thanks to its strong miscibility with that fuel. It can be combined with diesel to form a vehicle mixture [4, 5]. The practice of mixing B20 (Biodiesel with petroleum diesel) is widespread around the world. It is challenging because of the crucial requirements for reducing manufacturing costs, choosing suitable feedstock, and choosing a catalyst for biodiesel production. Transesterification of fats and oils is a typical method of producing biodiesel. This technique converts the triglycerides to monoglycerides, diglycerides, and straight-chain fatty esters. Biodiesel costs mainly depend on the oil and catalyst used to complete the transesterification step [6].

Biodiesel can be produced from both edible and non-edible oils. However, using edible oil is not a practical option because it might quickly lead to a shortage of oil feedstock due to overuse. Recycling and reusing waste products (such as oil and catalysts) can be managed through reverse logistics. Waste product utilization has become necessary in today's world and prompted the hunt for less expensive and more accessible raw materials, primarily interested in inexpensive, non-edible oils such as karanj oil, kusum oil, mahua oil, residue oil, rice bran non-edible oil, kapok oil, neem oil, and waste cooking oil among others. Most communities in developing nations throw UCO into the environment. It seriously harms the environment, society, the economy, and health. When cooking oil is dumped into water bodies illegally or inadequately, the quantity of organic pollutants in the water increases. As a result, the water quality is significantly degraded, endangering local communities, fish stocks, and other aquatic animals [7, 8].

UCO produces biodiesel in an environmentally friendly manner by recycling UCO and producing clean, renewable energy. Biodiesel can be made from spent cooking oil in a beneficial way from an economic, environmental, and waste management perspective [9]. A fat or oil reacts with an alcohol during the transesterification process to create esters and glycerol. Using a catalyst increases the reaction rate and yield, and when excess and low alcohol is added to it, the yield is low. The transesterification process can use a variety of alcohols, including methanol, ethanol, propanol, butanol, and amyl alcohol. The most widely used alcohols are methanol and ethanol due to their inexpensive cost, polarity, and short chain, among other physical and chemical advantages. In alcohol, as a catalyst, potassium hydroxide is generally used because it dissolves in no time. Alcohols, including methanol, ethanol, propanol, butanol, and even amyl alcohol, can all be used in the transesterification process. Methanol and ethanol are the most extensively utilized alcohols because they have several physical and chemical benefits over other alcohols, including their low cost, polarity, and short chain. Potassium hydroxide dissolves quickly in it, and it reacts rapidly with triglycerides. There is a required molar ratio of alcohol to triglycerides for transesterification to proceed stoichiometrically. Catalysts might be alkalis, acids, enzymes, nanocatalysts, or anything else that speeds up the reaction [10, 11].

Carbonates, sodium, and potassium alkoxide, sodium methoxide, sodium ethoxide, and sodium peroxide are all examples of alkali catalysts. It is common practice to utilize acid catalysts such as sulfuric acid, sulfonic acid, and hydrochloric acid. Since alkali-catalyzed transesterification proceeds significantly faster than acid-catalyzed transesterification, it is the method of choice in industrial settings. For best results, triglycerides must have a low free fatty acid concentration and less water [12]. The co-product, glycerine, is necessary to recover because it can be used as an industrial chemical and in products like dynamite, USP, and CP glycerol. By using centrifugation or gravity settling, glycerol can be separated. Base-catalyzed biodiesel synthesis typically uses potassium hydroxide as a catalyst [13, 14].

Nanotechnology has recently been employed to create nanocatalysts for the generation of biodiesel. Since most particle surfaces can be used for catalysis, nanoparticle catalysts are highly active [15]. Nanoparticles can be added to fuel to improve most of its qualities. Using nanoparticles in biofuel manufacture stabilizes chemical reactions and lowers biofuel production costs. In diesel engines, the use of nanoparticles dramatically raises brake thermal efficiency and lowers brake-specific fuel consumption. The toxic exhaust emissions released by diesel engines are reduced by nanoparticles [16]. Nanoparticles with particle diameters not more than 100 nm and a variety of forms and morphologies are used to create nanocatalysts. Since they exhibit the advantages of both heterogeneous and homogeneous catalysts, such as activity, selectivity, efficiency, and reusability, they can be thought of as existing at the crossroads of heterogeneous and homogeneous catalysis. The nanocatalyst, being positively charged, acts as an acid site. Adsorption of triglycerides to the acid sites of the nanocatalyst initiates the methanol-triglyceride reaction, which then proceeds rapidly on the nanocatalyst in a moderate environment [17, 18]. When methanol reacts with the primary nanocatalyst, it forms methoxide anions and hydrogen cations during the transesterification reaction. FAMEs, often known as biodiesel, are created when methoxide anions react with a triglyceride molecule in oil [19].

The work's primary goal is to increase the creation of fatty esters from waste products like UCO feedstock by utilizing nano-sized CaO catalyst particles that have been synthesized with high purity using the sol-gel process, and methods like XRD and SEM have been used to characterize. The reaction conditions were optimized for the synthesis of methyl ester.

Materials and Methods

Materials

UCO from a neighboring restaurant on the Andhra University campus in Vishakhapatnam (Andhra Pradesh, India) and CaO nanocatalysts were used in this work. To make the nanocatalyst, we purchased the necessary chemicals from a nearby chemical store [20]. The collected UCO was filtered using Whatman filter paper to separate the unwanted material before being heated to (110 ± 5 °C) for moisture removal. The filtered UCO still contains very few water molecules. Table 1 describes the physical characteristics of UCO, including color, viscosity, density, physical state, moisture level, and acid value.

Catalyst preparation

Using nanotechnology, CaO nanocatalysts were made using the sol-gel technique. A sol was made by dissolving calcium nitrate tetrahydrate in 25 ml of ethylene glycol in a 250 ml conical flask, which was then left unchanged for 5 h to form a gel. The gel is filtered with distilled water using Whatman filter paper after being motionless for 5 h. Because this process is reversible, the filtered gel is constantly dried in a hot air oven until completely dry. Using a mortar and pestle, the dried gel is ground into a fine powder. That dried powder was calcinated in a muffle furnace at (850 ˚C) for 1 h and stored in a desiccator for experimental purposes. The various sizes of catalysts were obtained.

Catalyst characterization

The samples underwent many characterization tests to evaluate the synthesized catalyst's characteristics [21, 22]. SEM was used to examine the samples' surface structure. XRD examination for the determination of crystalline sizes. Table 2 shows the physiochemical properties of nanocatalysts.

Results and Discussion

Effects of parameters

Transesterification was used to create biodiesel, and the procedure included mixing alcohol with catalyst and triglycerides in a reactor for a reaction and then separating and washing methyl ester and glycerol [23, 24]. The following variables were changed throughout the optimization process:

- Molar ratio of triglycerides to methanol.
- Catalyst weight.
- Reaction time.
- Reaction temperature.

Details about how the parameters mentioned above affect biodiesel production are provided in the following section. Each parameter can be altered to produce varying amounts of biodiesel [25, 26]. In this investigation, we use the transesterification technique with varying parameters, such as the molar ratio of triglycerides to methanol (from 1:5 to 1:10), the catalyst weight/volume percent (from 0.5% to 1.5%), the reaction time (from 80 min to 100 min), and the reaction temperature (from 40 ˚C to 60 ˚C) [27, 28]. Figure 1 depicts the relationship between reaction temperature and yield, figure 2 depicts the relationship between catalyst concentration and yield, figure 3 depicts the relationship between the oil-to-methanol ratio and yield, and figure 4 depicts the relationship between reaction time and yield.

Table 1: Physiochemical properties of UCO.

S. No.	Property	Value
1	Physical state	Liquid
2	Color	Thick brown
3	Kinematic viscosity	5.89
4	Acid value	0.4
5	Density	880
9	Specific gravity	0.88

Table 2: Physiochemical properties of nanocatalysts.

Product	CaO Nanopowder
Molecular formula	CaO
Appearance	White
Morphology	Spherical
Density	3.34 g/cc
Thermal conductivity	19.50WmK^{-1}
Melting point	2613 ˚C
Boiling point	2850 ˚C

Biodiesel production and characterization

The UCO was converted into methyl ester using a transesterification process. A 500 ml three-necked round-bottomed flask, a controlled magnetic stirrer with a hot plate, and a temperature sensor were used to conduct the transesterification process. First, 110 ˚C of pre-heating were applied to UCO to remove moisture. The necessary reaction temperature was reached by pre-heating UCO. The reactor was filled with the calculated amount of methanol, and the catalyst was continually agitated until completely dissolved [29]. Next, we added the preheated amount of UCO for the necessary period to produce biodiesel. Atmospheric pressure and a 1500 rpm stirring speed were used for all transesterification processes [30, 31]. Within the flask, a thermometer was set up to monitor the temperature of the reaction as it progressed. Following the reaction, the mixture was transferred to a separating funnel

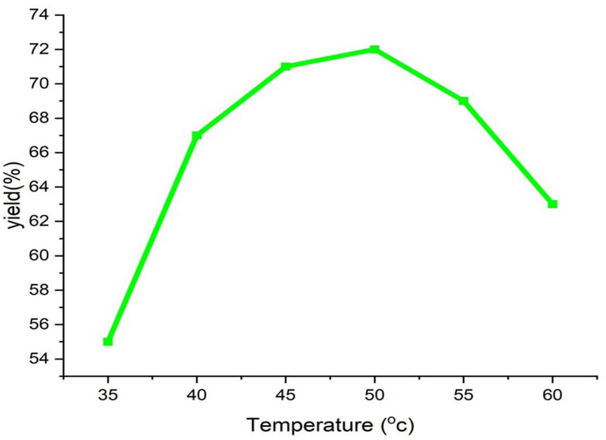

Figure 1: Temperature effect on biodiesel yield.

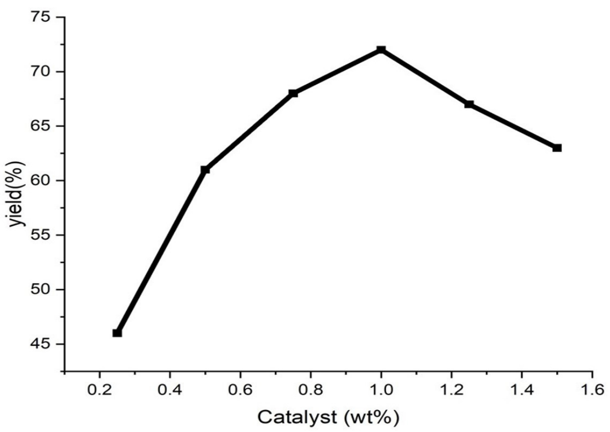

Figure 2: CaO catalyst's percentage impact on biodiesel yield.

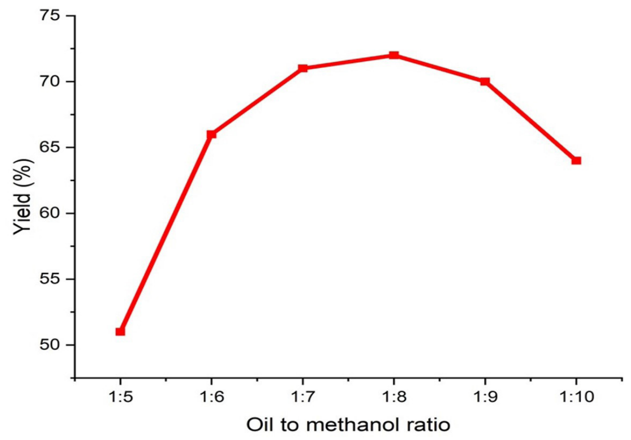

Figure 3: Effects of (oil-to-methanol) ratios on biodiesel yield.

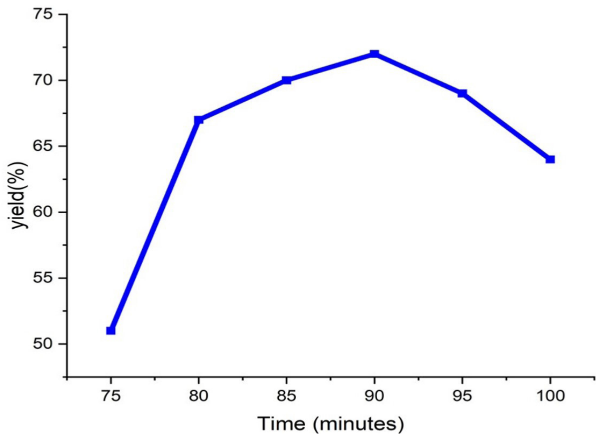

Figure 4: Reaction time (min) impact on biodiesel yield.

and rested overnight. Glycerol is denser than biodiesel, and the solid catalyst created three phases. Before separating in a separation funnel, it contains some excess methanol. To remove it was heated to above 65 °C for further uses, and the bottom layer, which contained glycerol, was tapped off using a separation funnel [32]. The generated biodiesel was then put in storage in anticipation of being examined further. From the equation, the outturn of biodiesel was determined.

Biodiesel yield = (mass of biodiesel produced/mass of UCO used) x 100

Protocols for testing the fuel (flash point, cetane number, and calorific value) and physicochemical (density, viscosity, moisture content, and acid value) qualities of manufactured biodiesel were developed by the Association of Official Analytical Chemists (AOAC) 1990. GC-MS analysis was used to identify the fatty acid composition in biodiesel [33].

Particularize biodiesel

The separated methyl ester was heated over methanol's boiling point (64.7 °C) to remove any additional unreacted methanol. The biodiesel's viscosity, specific gravity, acid value, density, our point, cetane number, flash point, and cloud point were tested per ASTM D 6751.1 after it had settled for two to three days to remove the extremely few suspended solid catalysts of the ASTM [34, 35]. The physicochemical properties of biodiesel are shown in table 3.

Table 3: Physiochemical properties of biodiesel.

S. No.	Property	Biodiesel value	ASTM standard values	Diesel value
1	Viscosity	4.23	1.9 - 6 $mm^{2/s}$	1.9 - 4.1 $mm^{2/s}$
2	Specific gravity	0.866	0.860 - 0.9	0.5
3	Density	866	860 - 900	
4	Flash point	135	93 minimum	60 - 80
5	Cloud point	5 + 6321	-3 to 12	-15 to 5
6	Pour point	-2.2	-15 to 10	-35 to -15
7	Acid value	0.53	0.8 maximum	0.24
8	Cetane number	54	47 minimum	40 - 55

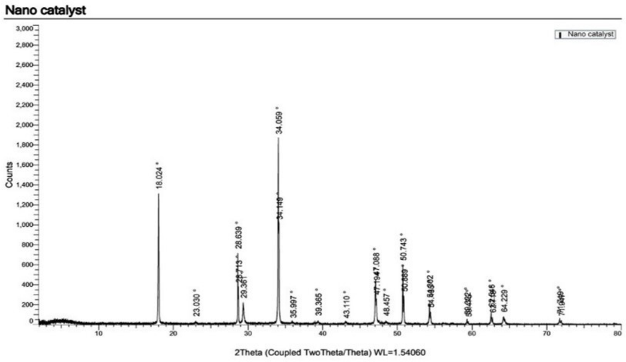

Figure 5: XRD pattern at the peak values as a result.

Catalyst characterization

XRD analysis

The primary elements of the synthesized catalyst were identified using XRD, which was also used to gauge the size of the crystallites [36]. The XRD investigation used electron high tension of 5 kV, wavelength dispersive X-ray fluorescence. figure 5 displays the XRD diffraction intensity (pattern) of CaO nanoparticles, and the value of the synthesized catalyst is revealed to lie between 17 and 73°; the sharp spectra reveal the powder's high crystallinity [12, 37]. The sharp peaks were visible in the peak position ranges of 18.02701, 28.64963, 34.07143, 47.10772, 50.76936, 54.32908, 62.57731, 59.35624 and 29.37647. The Debye-Scherer equation ($D = k\lambda/(\beta\cos\theta)$) was used to compute the CaO nanoparticle's crystallite size diameter (D), which is measured in nanometers (nm) [38]. As seen in table 4, the synthesized CaO has a mean crystal size of 47.60 nm and a particle size range of 24 to 79 nm.

SEM analysis

According to figure 6a to 6d, the SEM study was carried out at magnifications of 5 m, 10 m, 20 m, and 100 m. SEM pictures show that the produced CaO nanocatalyst consists of irregularly shaped particles with a porous structure and active sites [39]. On the other hand, the heterogeneous nature of the catalyst's particle sizes and shapes suggests that its reaction surface is more extensive. The SEM was used to investigate the structure of the obtained CaO nanocatalyst.

Table 4: XRD analysis of a synthesized nanocatalyst.

Peak position (2θ) in radians	Scherrer constant (κ)	Full width at half maximum (β) in radians	XRD radiation of wavelength (λ) in nanometers	Relative intensity (%)	Diameter of particles (nm)
18.02701	0.94	0.10696	0.154178	70.2	78.60
28.64963	0.94	0.11494	0.154178	38.3	74.56
34.07143	0.94	0.15305	0.154178	100	56.74
47.10772	0.94	0.23084	0.154178	13.7	39.24
50.76936	0.94	0.19901	0.154178	32	46.18
54.32908	0.94	0.24228	0.154178	6.7	38.52
62.57731	0.94	0.28145	0.154178	4.2	34.52
59.35624	0.94	0.27771	0.154178	2.8	34.42
29.37647	0.94	0.33504	0.154178	11.7	25.62

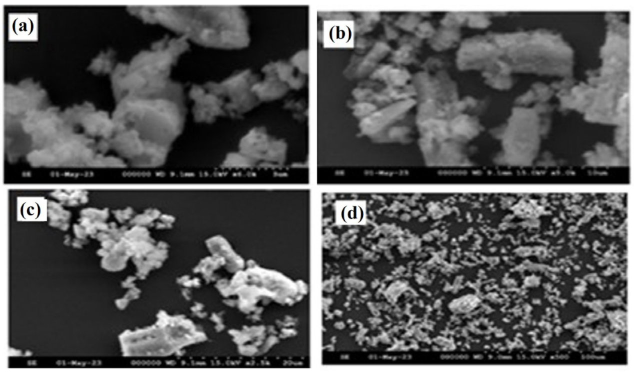

Figure 6: Shows the SEM images of synthesized nanocatalysts at 5 m, 10 m, 20 m, and 100 m magnification, respectively.

FTIR analysis

The absorption or transmission of an object can be determined, and an infrared spectrum is generated using the FTIR method. High-resolution spectrum data over a broad spectral range can be collected quickly and easily with an FTIR spectrometer. It measures the amount of light at each wavelength that a sample absorbs. One of the most straightforward techniques is "dispersive spectroscopy," which entails shining a monochromatic light beam on a sample, determining how much light is absorbed, and then repeating for each wavelength [40]. The range of the wavenumber is 500 to 4000 cm^{-1}. The FTIR spectroscopy pattern is shown in figure 7, and the functional groups are listed according to peaks in table 5. The figure 8 shows the reaction parameters of biodiesel vs yield (%).

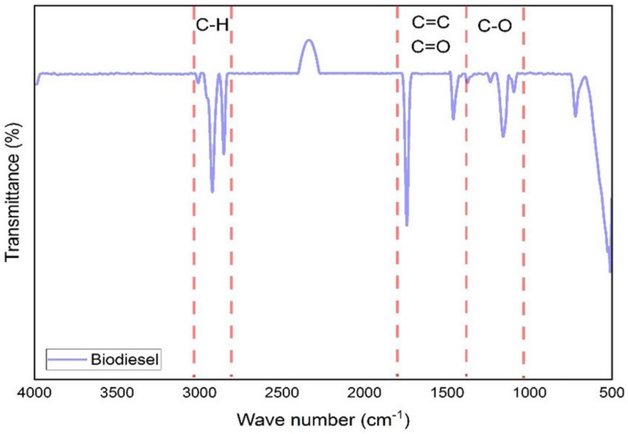

Figure 7: FTIR spectra of biodiesel.

GC-MS analysis

GC-MS is mainly used for the qualitative analysis of the fatty acid profile in UCO and in biodiesel [41]. As per the analysis, the methyl esters found in biodiesel are oxalic acid, butyl propyl ester, hexadecanoic acid, methyl ester, oxyranehexadecyl, 9,12-octadecadienoic acid, methyl ester, 9-octadecenoic acid, methyl ester, methyl stearate, oleic acid, glyidyloleate, and 9-oleate, which have both long and short carbon chains of single and carbon-carbon double bonds from C9 to C21, in which standard biodiesel's carbon chains range between C12 and C22. The fatty acid profile of biodiesel and UCO are shown in figure 9 and figure 10; the fatty acid profile of biodiesel and UCO, along with retention time, are shown in table 6 and table 7, respectively.

Table 5: FTIR studies of biodiesel.

S. No.	Wave number (cm^{-1})	Functional group	Functional group name	Transmittance (%)	Intensification
1	3008.51	O-H/C-H	Carboxylic acid/alkanes	82	Medium
2	2922.84	O-H/C-H	Carboxylic acid/alkanes	33	Medium
3	2853.03	O-H/C-H	Carboxylic acid/alkanes	28	Medium
4	1743.50	C=O	Esters/saturated aliphatic	13	Strong
5	1461.60	C-C/C-H	Aromatics/alkanes	77.4	Medium
6	1378.31	C-H	Alkanes	87.5	Medium
7	1237.38	C-O	Phenols	78.9	Medium
8	1150.88	C-O	Aliphatic amines	75.4	Medium
9	1003.35	N-H	Ammonium ions	88.3	Strong
10	721.40	C-H	alkanes	89.8	Medium

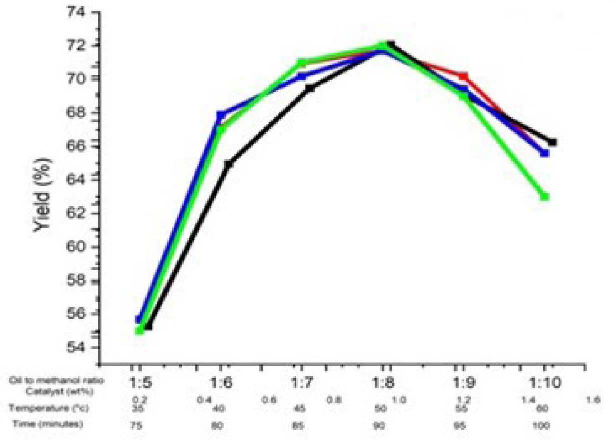

Figure 8: Reaction parameters for biodiesel yield (%).

Cost estimation

The selling price of biodiesel and production capacity were two production factors whose effects on gross profit were studied using sensitivity analysis. The input variables' sensitivity limits are set at 50 and + 50% from the base case. Compared to the cost of producing biodiesel, the production capacity has a high gradient [42]. We might infer that production capacity, as opposed to biodiesel price, significantly impacts gross margin more.

The cost evaluation of a chosen methyl ester of oil with a high free fatty acid content must be considered in the cost analysis of the obtained UCO methyl ester. The raw feedstock primarily drives the cost of producing biodiesel. The present values of raw materials and products were calculated in INR and their equivalents in USD; the value of these materials was calculated in August 2023, and the exchange rate was equivalent to (1 USD = 82.56 INR) shown in table 8. The cost of transesterification and cost analysis for biodiesel production are shown in table 9 and table 10.

One liter of UCO methyl ester yields 41 ml of glycerol during the transesterification process, and by recovering and selling the ingredients, we may create biodiesel from UCO with a 55% efficiency for as little as 45.23 INR equivalent to 0.55 USD.

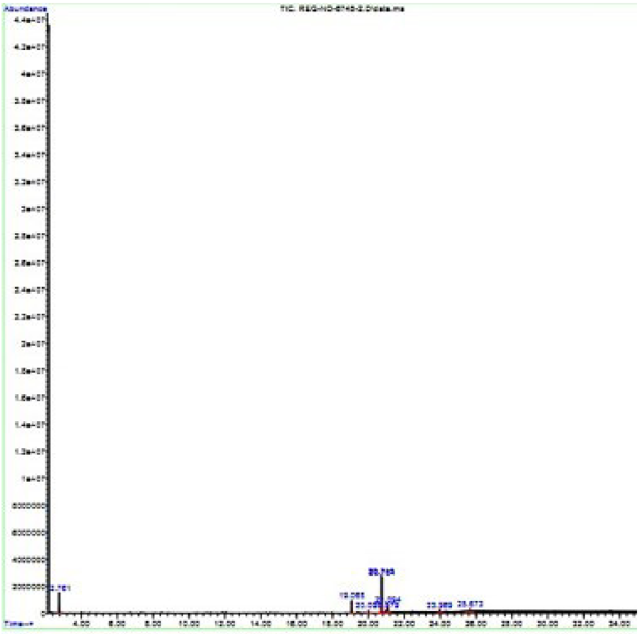

Figure 9: Shows the GC-MS of each fatty acid in biodiesel.

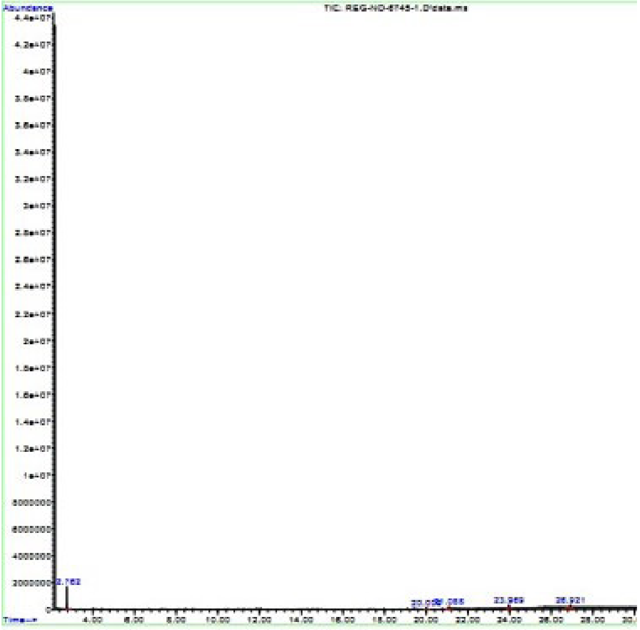

Figure 10: GC-MS of each fatty acid in UCO.

Table 6: The fatty acid profile of biodiesel.

S. No.	Fatty acid methyl ester	Molecular formula	Structure (C:D ratio)	Molecular weight	Retention time (min)
1	Oxalic acid, butyl propyl ester	$C_9H_{16}O_4$	9:2	188.22	10.72
2	Hexadecanoic acid, methyl ester	$C_{16}H_{32}O_2$	16:0	270.45	10.26
3	Oxirane, hexadecyl	$C_{18}H_{36}O$	18:0	268.47	2.06
4	9,12-Octadecadienoic acid, methyl ester	$C_{18}H_{32}O_2$	18:2	280.4	26.79
5	9-Octadecenoic acid, methyl ester	$C_{19}H_{36}O_2$	19:1	296.5	26.17
6	Methyl stearate	$C_{19}H_{38}O_2$	19:0	298.5	1.75
7	Oleic acid	$C_{18}H_{34}O_2$	18:1	282.5	1325
8	Glyidyloleate	$C_{21}H_{38}O_3$	21:1	338.5	4.13
9	9-Octadecenoic acid(Z)-2,3 dihydroxy propyl ester	$C_{21}H_{40}O_4$	21:1	356.5	4.86

Table 7: The fatty acid profile of UCO.

Fatty acid methyl ester	Molecular formula	Structure (C:D)	Molecular weight	Retention time (min)
Propanoic acid, ethyl ester	$C_5H_{10}O_2$	5:1	102.1317	2.756
Eicosanal-	$C_{20}H_{42}$	20:0	282.5	20.013
9-Octadecenoic acid, (E)-	$C_{18}H_{34}O_2$	18:1	282.4614	21.087
9-Octadecenal, (Z)-	$C_{18}H_{34}O$	18:1	266.5	23.965
Squalene	$C_{30}H_{50}$	30:0	410.7	26.919

Table 8: Present values of materials.

Materials	INR	USD
UCO	46 per liter	0.56 per liter
Methanol	28 per liter	0.34 per liter
Calcium nitrate tetrahydrate	620 per kg	7.51 per kg
Ethylene glycol	50 per liter	0.61 per liter
Sodium hydroxide	19 per kg	0.23 per kg
Biodiesel	77 per liter	0.93 per liter
Glycerol	85 per liter	1.03 per liter

Conclusions

To facilitate the transesterification of UCO into biodiesel, a CaO nanocatalyst with an average particle diameter of 47.60 nm was synthesized by sol-gel synthesis. With a 1:8 molar ratio of UCO to methanol, 1% by weight of CaO nanocatalyst, 50 °C reaction temperature, and 90-min reaction time, the highest biodiesel yield was 72%. The manufactured biodiesel's viscosity, specific gravity, density, flash and fire point, cloud and pour point, and acid value content were all reasonably compliant with the standard as tested in line with American fuel requirements (ASTM D 6571). The final cost estimation was 45.23 INR, equivalent to 0.55 USD per liter. UCO's biodiesel, regarded as renewable energy, is potentially useful as a cleaner alternative to diesel as a home energy fuel.

Acknowledgments

None.

Conflict of Interest

None.

Table 9: Cost of transesterification.

Materials	INR	USD
UCO	4.6	0.06
Methanol	1	0.01
Calcium nitrate tetrahydrate	0.62	0.01
Ethylene glycol	1.2	0.01
Sodium hydroxide	0.04	0.01

Table 10: Cost analysis of UCO methyl ester.

Processing input	INR	USD
UCO	46	0.56
Cost of filtering	10	0.12
Cost of transesterification	7.46	0.09
Total cost	63.46	0.77
Sale of by-products (glycerol)	15.42	0.19
Recovery materials (Methanol and catalyst)	2.81	0.03
Net cost of UCO methyl ester/liter	45.23	0.55

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